# Organic Chemistry Laboratory Major's Section CHEM 2473 Fall, 2007

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**Text**: "Microscale and Miniscale Organic Chemistry Laboratory Experiments", Second Edition, by Schoffstall, Gaddis, and Druelinger.

#### General:

Lab is scheduled from 2:00 to 5:30 on M afternoon. Please come, on time, to your assigned lab section. Attendance will be taken. Each lab will begin with a brief introduction to the experiment to be done that day.

The schedule of experiments for the quarter is attached. Please read the assigned sections in your lab book **before** coming to lab and do the "Prelab Assignments" that are specified in this syllabus. You will not be allowed to begin the experiment until the TA has verified that you have completed the "Prelab Assignments" and you have prepared your lab notebook for the experiment. If you read the assignments before lab and plan your time well, you will find it easy to complete the experiments in the allotted time or less. Planning ahead will also enable you to use your time most effectively. For example, an experiment might require that a reaction mixture be heated for one hour. The best use of your time would be to get the reaction started immediately. Then, while the reaction is being heated, you could distill the product from the preceding experiment, take melting points, wash glassware, or even study. In addition, you will find it advantageous to clean your glassware before you leave for the day. When you come for the next lab, your glassware will be clean, dry, and ready to use.

#### Safety:

#### **READ PP. 3-5 IN YOUR LAB TEXT.**

The organic laboratory is a potentially dangerous place. However, no accidents need occur if you are careful and are constantly aware of what you are doing and why. Read the directions given in each experiment carefully and in advance. Pay particular attention to the "Special Instructions" that are described for each experiment. Make sure you understand each step of the experimental procedure and any potential dangers. If you have questions, ask! Use your common sense and, above all, THINK!

### The following safety rules will be in effect at all times:

- 1. Safety glasses must be worn at all times.
- 2. Wear proper attire; gloves and lab coats when necessary; shoes (no sandals); no shorts.
- 3. No flames or smoking allowed in the lab.
- 4. No food or drink allowed in the lab.
- 5. No horseplay allowed in the lab.
- 6. Keep all work areas clean and orderly. This includes your bench, the reagent table and the hoods. Clean up all chemical spills immediately.
- 7. Use proper disposal procedures, as specified by your TA, for all chemicals and solutions.
- 8. Be careful to avoid contaminating the reagents. Close all containers snugly after use.
- 9. Be sure to read the labels on chemical containers carefully. Many chemical names are very similar.
- 10. Most organic chemicals are toxic to some extent. Treat them all with respect. Avoid getting them on your skin or clothing and avoid extensive breathing of their fumes. Work in the hoods when the experimental directions so instruct.
- 11. No students are allowed in the lab unless the TA is present. Only students assigned to that lab section are allowed in the lab. No student is allowed in the stockroom without explicit permission of the TA.
- 12. No chemicals, glassware or equipment are to be removed from the lab. No unauthorized experiments are to be done.

#### Notebooks:

You must have a bound lab notebook. You may use the same notebook and the same style as last quarter.

#### Prelabs:

Prelabs are to be done in the same manner as last quarter.

#### Reports:

Reports are to be done in the same manner as last quarter.

#### Products:

For synthesis experiments, submit the product in a clean, labeled vial along with the report. The label should have your name and the structure, weight, percent yield and mp or bp range of the product.

# Grading:

Your grade will be based on a total of 1020 points, distributed as follows:
Prelab Assignments, 10 pts./exp. 60 points
Lab reports and products (if applicable), 100 pts./exp. 900 points
Lab notebook, other than pre-lab write-ups 40 points
Subjective evaluation 20 points

# Schedule of Laboratory Experiments Major's Section Fall Quarter, 2007

Week of Sept. 10

Check-In

**Experiment 1** Reduction of Benzil

Read pp.364-366. Do Experiment 15.1A. Do Prelab Assignments 1 and 2. Include Critical Thinking Questions 1, 3 and 6 in your lab report. The report is due during the week of Sept, 17.

### Week of Sept. 17

# **Experiment 2** The Grignard Reaction

Read pp. 351-357. You will do a procedure very similar to Experiment 14.1A, except on a larger scale. The procedure for this experiment is available on Blackboard. Because it is so dry in Colorado, you do not have to specially dry your equipment if it has been drying in your drawer since the previous lab. Do Prelab Assignment 1. This is a two week experiment. You must get to the step where the benzophenone has been added to the Grignard reagent this week. You can complete the workup and isolation of the product next week if necessary. Include Critical Thinking Questions 1, and 2 in your lab report. The report is due during the week of Oct. 1.

### Week of Sept. 24

# **Complete Experiment 2**

# **Experiment 3** Combinatorial Chemistry and the Synthesis of Fruity Esters

Read pp. 430-436. Work in groups of three and do Experiment 20.1A today. Do Prelab Assignments 1-4. Next week do Experiment 20.1B. One person should do General Procedure 1 and another should do General Procedure 2. Design the experimental procedure before lab and have your procedure approved by the TA before you start. Include Critical Thinking Questions 1, 2, 3, and 6 in your lab report. The lab report is due during the week of Oct. 8.

# Week of Oct. 1 Complete Experiment 3

#### Week of Oct. 8

# **Experiment 4** The Wittig Reaction

Read pp.385-389. Do Experiment 17.1B. Analyze your product by TLC. Do Prelab Assignment 1. Include Critical Thinking Questions 1, 2, 4, and 5 in your lab report. The lab report is due during the week of Oct. 15.

#### Week of Oct. 15

#### **Experiment 5** Mixed Aldol Condensations

Read pp. 404-407 and 410-411. Do Experiment 18.1C. Work with a partner and each do the reaction of the unknown aldehyde with one of the ketones. Be sure to recrystallize your product twice. From the melting points of your two products you

should be able to identify the unknown aldehyde. Do Prelab Assignment 1. Include Critical Thinking Questions 3 and 6 in your lab report. The lab report is due during the week of Oct. 22.

#### Week of Oct. 22

# **Experiment 6** Synthesis of a Coumarin Derivative

Read pp. 441-445. Do Experiment 21.1B. The reaction requires a 2 hr. reflux period. Set up this reaction first. During the reflux period you will have time to do Experiment 7. Do Prelab Assignments 1 and 2. Include Critical Thinking Questions 2 and 8 in your lab report. The lab report is due during the week of Oct. 29.

### **Experiment 7** Simulated Identification of an Unknown

Read pp. 537-540 and 551-552. There is no Prelab Assignment for this experiment. This lab is a computer simulation of the real experiment you will do next week. You will be assigned two unknown organic compounds to identify on the computer. In order to identify each compound you must tell the computer which test to run on the unknown. The computer provides you with the result of the test. You then have to decide what test to run next. You continue this process until you have identified the compound. Since the "tests" on the computer can be done easily, this experiment allows you to hone your skills before undertaking the real thing. Make sure to interpret the IR and NMR spectra of your unknown if they are available. Record the results of each test just as though this is an experiment done in the lab. Your report should include a complete write-up of the identification of one unknown, just as thought the procedures were actually done in the lab. Also include the identity of the other unknown that you were assigned. The report is due during the week of Oct. 29.

#### Week of Oct. 29

# **Experiment 8** Identification of an Unknown

There is no Prelab Assignment for this experiment. This time you will be given a sample of a real unknown to identify. It will be an alcohol, aldehyde, amine, carboxylic acid, ketone, or phenol [one of the compounds listed in the tables in Appendix A (pp. 627-631)]. Follow the procedure outlined in Experiment 28.2 on pp. 537-577 in your lab text. You must do all 8 steps listed on p. 538, in addition to obtaining IR and NMR spectra of your unknown. This is a two-week experiment and the report counts twice the normal amount. The report is due during the week of Nov. 12.

# Week of Nov. 12 Clean Up, Check Out

You must check out of lab at this time, turn in your breakage card and pay for any equipment you have broken or you will receive an I as your lab grade. **Do not forget to turn in your lab notebook.**